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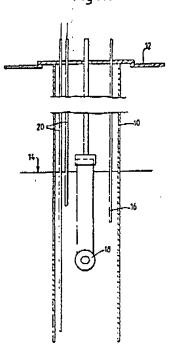
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(54) Monitoring the chemical composition of a fluid

(57) Apparatus for monitoring the chemical composition of a fluid eg liquid 14 having at least two components comprises sensors such as temperature probe 16, pressure dip tubes 20 for density measurement, and conductivity probe 18 for measuring at least two physical parameters dependent on the relative proportions of the components. The electrical measurements are processed by a micro processor using a series of polynomial equations relating the parameters with concentration and which are solved simultaneously by an iterative technique to derive values of the concentrations of the different components in the fluid. Control means may receive leedback signals so that any drift from a desired concentration can be corrected. The apparatus may be used in a plant for dissolving uranium ore in nitric acid, the components being acid and water, or in reprocessing irradiated nuclear fuel.

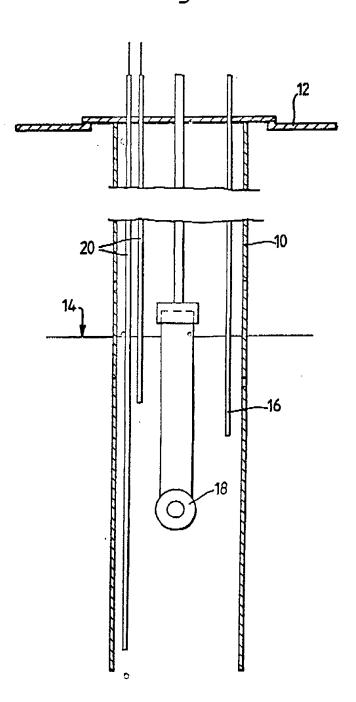


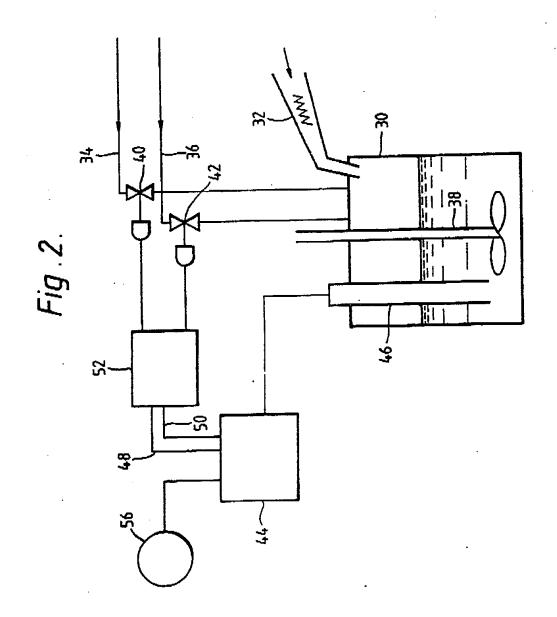
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The claims were filed later than the illing date within the period prescribed by Rule 25(1) of the Patents Rules 1982.

Fig . 1.





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Monitoring the chemical composition of a fluid

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This invention relates to the monitoring of the chemical composition of a fluid, eg a liquor, in which at least two components are present. The liquor may for example comprise an acid solution of a uranium salt as obtained in the course of reprocessing irradiated nuclear fuels in which case the role of the invention may lie in the monitoring of the acid and uranium concentrations so that appropriate confrol actions can be taken to optimise the process.

One method of determining acidity and concentration values is to use a titration technique but this suffers from the drawback that samples of the liquor have to be taken periodically and processing of the samples using titration techniques inevitably yields results some considerable time after taking the samples.

approach in which concentration measurements are made by measuring certain "global" physical parameters of the liquor, namely conductivity and specific gravity, these being parameters which are governed by the relative concentrations of the acid and uranium components of the liquor. The apparatus described in the prior Patent relied on the fact that, while the relationships between conductivity, specific gravity, acid and uranium concentration are not linear, linear approximations can be employed when working within a limited range centred

on predetermined concentration values and, in practice, a differently adjusted pair of resolver networks had to be employed for each working range. The known apparatus was also limited to a narrow range of temperatures (25 + 5°C), temperature compensation being affected by scaling 5 the electrical analogue signals corresponding to conductivity and specific gravity respectively in electrical networks incorporating resistance thermometers. There is also the major problem of interaction between the components in the system, which 10 is dealt with only in a very limited way by the resolver networks of Patent No 950198. Such interaction results in responses that are not additive; for example the conductivity of a solution containing a given uranium and free acid concentration, at a given temperature, is not 15 the sum of the conductivities of the two solutions containing the same concentrations of uranium and free acid separately.

The object of the present invention is to provide
improved monitoring of the chemical composition of a
fluid over extensive ranges of concentration and
temperature where the relationships may be non-linear
with complex interactions between the component
concentrations and/or temperature.

According to one aspect of the present invention there is provided apparatus for monitoring the chemical composition of a fluid, comprising sensor means for providing respective electrical measures of at least two

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physical parameters of the fluid, which parameters are dependent on the relative proportions of the components present in the system, means for converting said electrical measures into digital form and digital computing means for utilising the resulting digital values to derive, by an iterative technique, values of M_1 , M_2 . . . satisfying simultaneous equations of the form $f_N = g_N$ (M₁, M₂ . . .) $-P_N$ where $f_N = 0$ P_N is the digital value of the Nth parameter and g_N are second or higher order polynomial functions relating 10 the concentrations M_1 , M_2 . . . of the components in the fluid to the respective parameter value P_N and where the functions gN contain cross product terms, representing interactions, involving the concentrations M_1 , M_2 . .

According to a second aspect of the invention there is provided fluid processing plant in which the fluid comprises at least two components the concentrations of which are to be controlled, said plant including: means for controlling the introduction of at least one of the components so that the concentration of at least one of the components is thereby varied, sensor means for providing respective electrical measures of at least two physical parameters of the fluid, which parameters are dependent on the concentrations of the components present in the system, means for converting said electrical measures into digital form and

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digital computing means for utilising the resulting digital values to derive, by an iterative technique, values of M_1 , M_2 ... satisfying simultaneous equations of the form $f_N = g_N$ $(M_1, M_2 ...)$ - P_N where $f_N = 0$, P_N is the digital value of the Nth parameter and g_N are second or higher order polynomial functions relating the concentrations M_1 , M_2 ... of the components in the fluid to the respective parameter value P_N and where the functions g_N contain cross product terms, representing interactions, involving the concentrations M_1 , M_2 ...

Preferably the plant or apparatus further includes means for sensing the temperature of the fluid and providing an electrical measure of the temperature (t) for conversion into digital form by the converting means, the functions g_N in this case also including t as a variable.

In one example of the invention involving a two
component liquor, eg containing nitric acid and uranyl

20 nitrate, the equations can be of the form:

f1 = Co + C1M1 + C2M2 + C3t + C4M12 +

C5M22 + C6t2 + C7M1M2 + C8M1t +

C9M2t - P1

f2 = Ko + K1M1 + K2M2 + K3t + K4M12 +

K5M22 + K6t2 + K7M1M2 + K8M1t

. .(2)

 $+ K_9M_2t - P_2$

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where the coefficients Co - C9 and Ko - K9 are constants which may be derived using multiple polynomial regression by measuring the customary dependent variables P₁ and P₂ experimentally from a series of liquor 5 samples covering a wide range of known concentrations (M1, M2) of the components and known temperatures, M₁, M₂ and t being the customary independent variables. Although f1 and f2 are expressed above as second order polynomials, they may be expressed as third 10 or higher order polynomials depending on the system, eg second order polynomials have proved adequate for a nitric acid/uranyl nitrate system whereas third order polynomials have been found to be more appropriate for certain concentration ranges of a KF/KOH system.

Thus, having determined values for the co-efficients Co-C9 and Ko-K9, substitution of the digital values P1, P2 and t derived for a liquor with unknown component concentrations allows the two simultaneous equations to be solved iteratively for M1 and M2.

The iterative technique employed may involve the known Newton Raphson process but modified for the solution of simultaneous equations.

For a two component system, the digital computing means may include:



- a. digital storage means for storing the values of the coefficients of the equations f₁, f₂ which will be predetermined (by experiment) for a given system such as nitric acid/uranyl nitrate or KF/KOH;
- 5 b. means for registering the outputs P₁, P₂ derived from the sensor means and, if applicable, the temperature prevailing in the system;
 - c. means for holding provisional values X_1 , X_2 for M_1 , M_2 ;
- 15 F₁ and F₂;
 - e. means for combining the provisional values X_1 , X_2 (and, if applicable, the measured temperature) with said coefficients in accordance with the respective first derivatives of f_1 , f_2 with respect to M_1 , M_2 in turn and calculating the resulting values F_{11} ,
 - F₁₂ and F₂₁, F₂₂;
 - f. means for deriving from said values correction terms T_1 , T_2 of the form

 $T_1 = (F_2*F_12*F_1*F_22)/(F_11*F_22*F_21*F_{12})$

25 $T_2 = (F_1 * F_2 - F_2 * F_{11})/(F_{11} * F_2 - F_2 * F_{12});$

- g. means for applying the correction terms T_1 and T_2 to X_1 and X_2 respectively; and
- h. means for comparing the newly corrected X₁, X₂

 values with the previously held X₁, X₂ values and

 depending on whether or not the result of comparison is
 favourable either updating the holding means with the
 corrected values and initiating a further cycle of
 iterative operation by means (d)-(h) or providing an
 output to indicate completion of the iterative process

 with the last corrected X₁, X₂ values representing
 the interatively-derived final values for the
 concentrations M₁, M₂.

Thus, means (g) may be operable to form the sums

X₁ + T₁ and X₂ + T₂ whilst means (h) may effect

comparison by determining the extent to which

(X₁ + T₁)/X₁ and (X₂ + T₂)/X₂ deviate from

unity; for example, if these ratios lie within the range

0.9999 to 1.0001 the comparison may be designated

favourable whereas ratios outside this range may be

designated unfavourable.

The values of M1, M2 . . . may be employed in completing a feedback loop to allow the concentrations of the components to be maintained within predetermined limits. Thus, the controlling means is preferably supplied with feedback signals representing

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the values M1, M2 . . . so that any drift away from the desired concentration levels in the system is corrected for by appropriate operation of the controlling means.

In an alternative arrangement where feedback control is not required, means may be provided for activating some form of alarm or modifying operation of the plant (eg by closure or opening of valves or such like) in the event that the concentrations of the components deviate from preselected working ranges.

The "global" parameters sensed by the sensor means 10 may be density and conductivity but other parameters, such as refractive index, surface tension, viscosity, speed of sound, light absorbence/transmissivity output counts from a gamma absorptiometer, XRF spectrometer etc, may be employed depending on the fluid. As a general rule, for good results, of the parameters measured at least one should have a response to variations in component concentration which is significantly different for the components. For example, in the case of a fluid containing nitric acid/uranyl nitrate, density variations 20 are responsive predominantly to changes in uranyl nitrate concentration whereas nitric acid concentration changes tend to predominate in the case of conductivity variations.

To promote further understanding of the invention, examples of the invention will now be described with

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reference to the accompanying drawings in which:

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Figure 1 indicates diagrammatically a multi-probe sensor arrangement for sensing density, conductivity and temperature of a KOH/KF solution contained within a vessel forming part of a HF scrubber using KOH as scrubbing solution; and

Figure 2 is a schematic diagram showing a chemical plant employing a monitoring apparatus as part of a feedback loop for regulating concentration of a liquor.

protective vented tubular sheath 10 extending from the vessel top 12 and below the liquor level 14. The sheath 10 houses a solid state temperature probe 16, an electrodeless conductivity probe 18 and pneumicator tubes 20 forming part of a density-measuring instrument. The latter operates by pumping a suitable gas such as nitrogen through the dip tubes 20 whose lower ends are vertically staggered by a known distance and measures the difference in gas pressure, the density of the liquor being calculated from this difference.

The analogue outputs from the probes 16, 18 and the density meter are fed via interface circuitry including analogue/digital converter circuits to a microprocessor programmed to execute the mathematical routines that will be described below. These routines are based on the polynomial equations (1) and (2) set out hereinbefore.

As mentioned previously, the coefficients/constants involved in these equations must be derived

experimentally for a given system, for example by making up sample solutions containing a wide range of known concentrations of the components (ie covering at least the range that can be expected in practice) and measuring the "global" parameters, ie density and conductivity in this case, for each sample over a range of temperatures (again covering at least the range that can be expected in practice). The experimental results obtained for P₁, P₂ and the known concentrations M₁, M₂ can then be processed using multiple regression analysis to obtain "best-fit" values for the coefficients C₀, C₁
. . . and K₀, K₁ . . . These values then apply for the given system and can be stored as an array in a digital memory associated with a microprocessor.

In theory the C and K values can be derived by measuring n solutions if there are n values of C and K in each of the 2 equations. Each of the n solutions would have a different KOH and/or KF concentration and a different temperature.

In practice, more than n solutions are always
measured and each solution is measured at different
temperatures. This is convenient practically and gives
more precise determinations of C and K. (The smaller n
the less precise the determinations of C and K and hence
the less precise the subsequently measured component
concentrations).

Typically 20-40 solutions are measured, each at 5-20

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different temperatures covering the temperature range the plant solution will encounter. This results in hundreds of equations, which are solved for the C and K values using multiple polynomial regression. Selecting the concentrations of the 20-40 solutions is done as follows. The concentration range of each component occurring in the plant liquor is recorded and then extended somewhat at the lower and upper end. Each continuous range is then divided, typically, into 10-20 discrete concentrations. Each discrete concentration of one component is then linked either a) at random or b) in a systematic manner, depending on the application, to a discrete concentration of another component. Constraints are frequently put on this linking. These constraints are either a) a combination of component concentrations which is physically impossible or b) a combination which

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For the HP scrubber, the 2 components are KOH and KF. The KOH absorbs HF which is converted to KF.

could not occur on the plant.

- Thus the KF concentration rises as the KOH concentration falls. For example, 28 solutions initially measured to determine the values of C and K were chosen systematically as follows, each solution being measured over the range 5-40°C at approximately 5°C increments.
- The conductivity and density of each solution was measured at the 8 different temperatures giving 224 equations relating conductivity to KOH and KF

concentration and temperature, and similarly for density.

For a third order polynomial there are 19 values of C to

determine (conductivity) and 19 values of K (density).

The solutions are always measured in a random order, to

SOLUTION	gKOH/100ml	gKF/100ml
1	16.8	О .
2	14.0	2.9
3	11.2	5.8
. 4	8 .4	8,7
5	5 .6	11.6
6	2.8	14.5
7	$\mathfrak{c}_{\!\scriptscriptstyle \mathcal{O}}$	17.4
8	14.0	0
9	11.2	2.9
10	e.4	5.8
11	5 .6	8.7
12	2.8	11.6
13	Q	14.5
14	11.2	0
15	8.4	2.9
16	5 .6 •	5.8
17	2.8	8.7
18	0	11.6
19	8 4	Ó
20	5 .6	2.9
21	⊉.8	5.8
22	0	8.7
23	5 .6	O
24	2 .8	2.9
25	o ·	5.8
26	2.8	0
27	0	2.9
28	0	0

avoid systematic errors.

C and K values are determined by multiple polynomial regression, using the method of least squares. In theory only 19 equations are required (for a third order polynomial system) to determine the 19 C values, and

overdetermined system, ie more equations than 'necessary' and has the advantage of providing the uncertainty in the values of C and K, ie expressed as standard deviations.

The method of least squares is typically used for solving

The microprocessor is connected to receive the outputs of the density, conductivity and temperature sensors via a suitable interface including analogue-to-digital conversion circuitry so that, in practice, the concentration levels in the liquor can be monitored by way of the measurements available for the density, conductivity and temperature.

In practice for a KOH/KF system, third order

15 polynomial equations have been used and the
microprocessor is programmed to execute the stepwise
procedure outlined below.

The following terms are used in the equations that follow:

20 y Conductivity (mS.cm⁻¹), as measured

overdetermined systems.

- w Density (g.cm⁻³), as measured
- t Temperature (deg C), as measured
- x KOH concentration (g KOH/100ml)
- z KF concentration (g KF/100ml)

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- A Calculated conductivity
- B Calculated density



- D Difference of calculated and measured conductivity
- E Difference of calculated and measured density
- P 1st differential of conductivity w.r.t. x
- 5 R 1st differential of conductivity w.r.t. z
 - Q 1st differential of density w.r.t. x
 - s 1st differential of density w.r.t. z
 - F Correction term for conductivity
 - G Correction term for density
- 10 E1 Error value for conductivity
 - E2 Error value for density

The above information is stored in memory arrays associated with the microprocessor to allow the intermediates to be printed at the end of a calculation.

- 15 The coefficients for the conductivity and density equations are held in arrays C(i) and K(i) respectively. Initially, arbitrary values are selected for x and z. The temperature to is conveniently the difference between a centred value for the temperature (ie the median of the operating temperature range of the plant) and the liquor temperature as measured.
 - Step 1 The values for x, z (initially arbitrary) and t are fed into the following equations to arrive at a calculated value of the conductivity (A) and density (B).

$$A = C_{1}x + C_{2}z + C_{3}t$$

$$+ C_{4}x^{2} + C_{5}z^{2} + C_{6}t^{2} + C_{7}xz + C_{8}xt + C_{9}zt$$

$$+ C_{10}x^{3} + C_{11}z^{3} + C_{12}t^{3} + C_{13}x^{2}z + C_{14}x^{2}t$$

$$+ C_{15}xz^{2} + C_{16}xt^{2} + C_{17}z^{2}t + C_{18}zt^{2} + C_{19}xzt$$

$$B = K_{1}x + K_{2}z + K_{3}t$$

$$+ K_{4}x^{2} + K_{5}z^{2} + K_{6}t^{2} + K_{7}xz + K_{8}xt + K_{9}zt$$

$$+ K_{10}x^{3} + K_{11}z^{3} + K_{12}t^{3} + K_{13}x^{2}z + K_{14}x^{2}t$$

$$+ K_{15}xz^{2} + K_{16}xt^{2} + K_{17}z^{2}t + K_{18}zt^{2} + K_{19}xzt$$

Step 2 - The difference between the derived and experimental conductivity and density

measurements is calculated.

$$D = A - y$$
 [= f_y (x,z)] Conductivity
 $E = B - w$ [= f_w (x,z)] \cap Density

The y and w equations contain the unknowns x and z, and are solved for x and z using a Newton Raphson iterative process adapted for the solution of simultaneous equations.

Step 3 - The equation for A is differentiated first w.r.t. 'x' and then w.r.t. 'z' to give the respective first differentials P and R.

$$+ 2C_{4}x + C_{7}z + C_{8}t$$

$$+3c_{10}x^{2} + 2c_{13}xz + 2c_{14}xt + c_{15}z^{2} + c_{16}t^{2} + c_{19}zt$$

$$S R = C_2$$

$$+ 3C_{11}z^2 + C_{13}x^2 + 2C_{15}xz + 2C_{17}zt + C_{18}t^2 + C_{19}xt$$

Step 4 - The equation for B is differentiated first

w.r.t. 'x' and then w.r.t. 'z' to give the

respective first differentials Q and S.

$$Q = K_1$$

$$+ 2K_{4x} + K_{7z} + K_{8t}$$

15 +
$$3K_{10}x^2$$
 + $2K_{13}xz$ + $2K_{14}xt$ + $K_{15}z^2$ + $K_{16}t^2$ + $K_{19}xt$

$$S = K_2$$

$$+ 2K_{5}z + K_{7}x + K_{9}t$$

$$20 + 3k_{11}z^{2} + k_{13}x^{2} + 2k_{15}x^{2} + 2k_{17}z^{2} + k_{18}t^{2} + k_{19}x^{2}$$

Step 5 - The correction term for the KOH and KF concentrations are calculated.

$$F = (E*R - D*S) / (P*S - Q*R)$$
 KOH concentration

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$$G = (D*Q - E*P) / (P*S - Q*R)$$
 KF concentration

Step 6 - The correction terms are now added to the



previous arbitrary values of the KOH and KF concentrations.

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$$x_{i+1} = x_i + F$$

$$z_{i+1} = z_i + G$$

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Step 6 - The error between consecutive calculations is calculated.

$$E1 = x_{i+1} / x_i$$

$$E2 = z_{i+1} / z_i$$

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Steps 1 to 6 are then repeated each time using the corrected values for x and z until E1 and E2 lie between for example 0.9999 and 1.0001. The values for x and z then prevailing are then taken as the KOH and KF concentrations existing in the liquor at that time and at the particular temperature measured.

In this manner, the KOH and KF concentrations can be continuously monitored and control of the system may be modified according to requirements. For example, the rate of supply of KOH to the scrubber may be controlled in dependence upon the concentration levels calculated so as to maintain the KOH/KF concentration levels within predetermined limits.

Referring now to Figure 2, this shows

25 diagrammatically plant for dissolving uranium ore in nitric acid, the ore being introduced into vessel 30 at a constant feed rate by means of a screw feeder 32 and the



acid being introduced via supply line 34 along with water fed via supply line 36. The contents of the vessel are agitated by means of stirrer 38. The supply lines 34, 36 include motorised flow-setting valves 40, 42 for regulation of supply of acid and water to the vessel 30 in order to maintain the concentrations of uranium and nitric acid within preset limits.

To control the valves 40, 42, the concentration of the liquor is monitored by means of a microprocessor-based analysis unit 44 which receives input signals from a probe assembly 46 which may be of the form shown in Figure 1. Thus, the probe assembly 46 provides electrical signals representing the density, conductivity and temperature prevailing in the liquor. The signals are processed by the analysis unit 44 in the manner described above to provide two analogue output signals which represent respectively the uranium and acid concentrations of the liquor and are applied via inputs 48, 50 to a programmable controller 52 (such as a Model 6433 Programmable Signal Processor marketed by Turnbull Control Systems Limited). The controller 52 is programmed to process the input signals to derive 3-term output control signals for operating the valves 40, 42 so as to maintain the flow rates of acid and water at values such that the uranium and acid concentrations remain within limits programmed into the controller 52.

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In addition to providing signals representing concentration levels to the controller 52, the analysis unit 44 is also connected to an alarm 56 and is programmed to actuate the alarm in certain circumstances, eg if a fault develops in the unit 44 or the probe assembly or if the concentration of either of the species being monitored falls outside preset limits.

Although the invention is described above in relation to specific applications, is KOH/KF

10 concentration levels in a HF scrubber or uranium/acid concentration levels in a dissolver, it will be understood that the same hardware can be used on different plant if the microprocessor is reprogrammed appropriately.

The invention is also applicable to systems in which there are three or more interacting components. Also the ambit of the invention is not restricted to liquors; gas, vapour and liquid systems are all considered to be within the scope of the invention. The parameters measured may be entirely different physical entities such as density and conductivity but they may be different characteristics of the same physical entity, egultraviolet absorption at different wavelengths.



Claims

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- Apparatus for monitoring the chemical composition of a fluid comprising sensor means for providing respective electrical measures of at least two physical parameters of the fluid, which parameters are dependent on the relative proportions of the components present in the fluid, means for converting said electrical measures into digital form, and digital computing means for utilising the resulting digital values to derive, by an iterative technique, values of M1, M2 . . . satisfying simultaneous equations of the form $f_N = g_N$ (M₁, $M_2 \cdot \cdot \cdot) - P_N$ where $f_N = 0$, P_N is the digital value of the Nth parameter and g_N are second or higher order polynomial functions relating the concentrations of the components M_1 , M_2 . . . in the fluid to the respective parameter value P_{N} and where the functions gn contain cross product terms, representing interactions, involving the concentrations M1, M2 . .
- 20 2. Apparatus as claimed in Claim 1 further including means for sensing the temperature of the fluid and providing an electrical measure of the temperature (t) for conversion into digital form by the converting means, the equations g_N in this case also including t as a
 - 3. Apparatus as claimed in Claim 1 or Claim 2 in which said digital computing means comprises:

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variable.



- a. digital storage means for storing the values of the coefficients of the polynomials f₁, f₂ which will be predetermined (by experiment) for a given fluid such as nitric acid/uranyl nitrate or KF/KOH;
- b. means for registering the outputs P_1 , P_2 derived from the sensor means and, if applicable, the temperature prevailing in the system;
 - e. means for holding provisional values X_1 , X_2 for M_1 , M_2 ;
- neans for combining the provisional values X₁,

 X₂ (and, if applicable, the measured temperature)

 with said coefficients in accordance with the

 respective polynomial functions f₁, f₂ and

 calculating the resulting values of f₁, and f₂,

 viz F₁ and F₂;
 - e. means for combining the provisional values x_1 , x_2 (and, if applicable, the measured temperature) with said coefficients in accordance with the respective first derivatives of f_1 , f_2 with
- respect to M_1 , M_2 in turn and calculating the resulting values F_{11} , F_{12} , F_{21} and F_{22} ;
 - f. means for deriving from said values correction terms \mathbf{T}_1 , \mathbf{T}_2 of the form
- 25 $T_1 = (F_2 + F_1 + F_2 + F_3)/(F_4 + F_5 + F_5)$ $T_2 = (F_1 + F_2 + F_3)/(F_4 + F_5 + F_5)$



- means for applying the correction factors T_1 and T_2 to X_1 and X_2 respectively; and
- h. means for comparing the newly corrected X₁, X₂

 values with the previously held X₁, X₂ values and

 depending on whether or not the result of comparison is
 favourable either updating the holding means with the
 corrected values and initiating a further cycle of
 iterative operation by means (d)-(h) or providing an
 output to indicate completion of the iterative process

 with the last corrected X₁, X₂ values representing
 the interatively-derived final values for the
 concentrations M₁, M₂.
- 4. In combination, apparatus as claimed in any one of Claims 1 to 3 and means operable to control the quantity of at least one of the components present in the fluid, the control means being supplied with feedback signals from the monitoring apparatus so that any drift away from the desired concentration levels in the fluid can be detected by the monitoring apparatus and corrected for by appropriate

 20 operation of the control means.
 - 5. Apparatus for monitoring the chemical composition of a fluid substantially as hereinbefore described with reference to, and as shown in, the accompanying drawing.

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6. Fluid processing plant including apparatus a claimed in any preceding claim, and including means for controlling the introduction of at least one of the components so that the concentration of at least one of the components is thereby varied.

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